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DEVELOPMENT OF HIGH TEMPERATURE RESISTANT MATERIALS OF USE IN N--ETC(U)  
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DEVELOPMENT OF HIGH TEMPERATURE RESISTANT  
MATERIALS FOR USE IN NAVAL ORDNANCE

Contract No. N00017-73-C-4306

Fourth Quarterly Report

Submitted to:

Naval Ordnance Systems Command  
Department of the Navy  
Washington, D. C. 20360

Prepared by:

ATLANTIC RESEARCH CORPORATION  
5390 Cherokee Avenue  
Alexandria, Virginia 22314

Bruno J. Macri

Period Covered:

6 April 1973 to 5 July 1973

18 July 1973

TR-PL-10124-00-4

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ALEXANDRIA, VIRGINIA 22314

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Pyrostrand® graphite composite is a carbon filament reinforced pyrolytic graphite formed by simultaneous pyrolytic graphite deposition and filament addition. A previous program<sup>(1)</sup> demonstrated attractive erosion resistant characteristics for rocket nozzle applications. Additional features of this concept include: (1) the simultaneous deposition of carbides (when desired) to modify the matrix structure, (2) the use of filamentary materials of various textile configurations and mechanical properties and (3) the control of reinforcement placement and content for increased strength and greater erosion resistance in very-high-temperature environments.

The pyrolytic graphite matrix has inherently high resistance to erosion under rocket motor firing conditions. A disadvantage of the material is its tendency to delaminate at moderate or excessive thicknesses. This delamination tendency can be reduced by causing the pyrolytic graphite to grow from surfaces other than the substrate, a filamentary reinforcement for example. The high performance graphite yarns have a very high strength/density ratio and thereby offer the potential for producing composites with a significant degree of reinforcement. In contrast to most materials, the strength of both major components of these composites increases with temperature up to near the sublimation temperature of graphite (6,600°F). The graphite fiber reinforcement sufficiently reduces the anisotropy of the pyrolytic graphite matrix so that freedom from delaminations of the pyrolytic graphite is achieved.

Rocket motor nozzle tests in 1-inch throat sizes have shown the applicability of these graphite composite materials (Pyrostrand) to the nozzle conditions of a typical advanced propulsion system. Results to date indicate that, under test conditions of 6500°F and 1000 psi, the Pyrostrand graphite composite erodes at an average rate of  $2 \pm 1$  mils per second.

Based on the expected erosion rate of approximately 2 mils per second, plus the high potential hoop strength, the Pyrostrand graphite composite nozzle appears to be a desirable throat insert material for these rocket motor conditions. Other nozzle candidates have major disadvantages. For example, tungsten has high weight and vulnerability to nuclear damage,

pyrolytic graphite coatings have a maximum thickness capability below that required for the severe C-4 motor conditions and bulk graphites or ablative carbon composites erode excessively. The Pyrostrand graphite composite materials are structurally capable of self support, due to the filamentary reinforcement.

Past efforts have been limited by furnace size and a small capacity for handling the filamentary material. The results were sufficiently promising to indicate that further work should be conducted to scale-up the equipment for the production of larger pieces.

## 2.0 WORK ACCOMPLISHED

During this reporting period, work accomplished related primarily to completing the design of the 18-inch furnace and the purchase of accessory components.

Construction of the 18-inch furnace involves four major sections: (1) the atmospheric control chamber, (2) the induction coil, (3) the furnace assembly and (4) the closed water system. Company funds were used for the purchase of the above mentioned equipment.

The atmospheric control chamber is a vacuum tank 72 inches in diameter and 60 inches high. It was constructed from 304 stainless steel in two sections, split horizontally, and provided with flanges for vacuum sealing. Operations under either vacuum or differential pressure conditions will be possible. A system will be installed to insure an adequate pressure release in the event of an inadvertent overpressurization. In addition, a pressure sensing device will be installed within the furnace to warn of sudden inadvertent pressure changes.

The lower section of the vacuum chamber contains three ports so that the winding of yarn on the mandrel may be observed from different vantage points during a run. All utility connections will pass through the tank wall.

During routine operations, the lower half of the tank will remain fixed in place and only the upper tank lid will be removed for access to the furnace area.



The induction coil was received in mid-May. It has been electrostatically sprayed with epoxy resin 10 - 12 mils thick, and lined with multiple layers of fiberglass and refractory cement. The as-received heating coil assembly is 33-inches ID, 41-inches high (stud board length), and the coil length - 36-inches high. The top 3 coil turns (6.5-inches) are for cooling only.

The furnace structure will be based upon the 8-inch chamber model presently in use, plus new concepts made possible by the increased size. The maximum furnace chamber diameter was selected to be 19-inches to accommodate nozzle inserts with a throat diameter of 12-1/2 inches. The dimensions of the coil and insulation were proportioned on this basis.

To promote efficient deposition of pyrolytic graphite, the free volume between sample and chamber wall must be kept relatively small. Fortunately, with graphite susceptors, the chamber diameter may be controlled by varying the wall thickness. The depth of penetration of graphite by the induced current of a magnetic field of 180 Hz is approximately 4-1/2 inches. Any wall thickness up to this value will allow the susceptor to operate efficiently. A 4-inch thick susceptor plus a 1/2-inch liner will provide a suitable chamber for the initial furnace studies.

A cooling plate on the bottom of the furnace will be bolted to the coil assembly and support the susceptor, work load and lampblack insulation. It will be possible to lift the entire furnace out of the bottom tank section. There will, however, be connections to be taken apart to remove the furnace.

Redundancy has been designed into the furnace control and cooling systems. Control flow meters, as well as visible flow indicators, are attached to each injector water cooling line. In the event of a water leak, that specific defective system can be shut off without damaging the other components by complete water shut-off. All outlet water temperatures will be monitored. The stainless steel base plate is cooled by two independent cooling coil lines which have separate control systems.

A closed water system for use with the vacuum furnaces is nearing completion. This system will permit adequate cooling water capacity independent of city water. However, in the event of an electrical power failure,

city water will be automatically directed into the recycling system.

Furnace support systems, such as gas dryers, plumbing and control panel installation, are nearing completion. Figure 1 shows the schedule for completion of the installation of the new 18-inch Pyrostrand furnace.

3. FUTURE WORK

During the next three months, the furnace will undergo check-out tests, followed by winding of Pyrostrand components. In accordance with verbal instructions, Atlantic Research and Battelle personnel will then establish the best direction for this Pyrostrand program. An initial meeting was held on 22 June 1973 at Atlantic Research with William Pfeifer and Stewart Veeck of Battelle. The intent is to insure that this program is consistent with the goals of the overall Navy Materials Development Effort.

REFERENCES

1. Eugene L. Olcott, Atlantic Research, "Development of High Temperature Resistant Materials for Use in Naval Ordnance (U)," Summary Report prepared under Contract No. N00017-71-C-4401 for Naval Ordnance Systems Command, July 1971. CONFIDENTIAL.



	June	July	Aug	Sept
Fabricate Carbon Components		—		
Closed Water System (Complete)		—		
Furnace Assembly (Complete)		—		
Furnace Test Runs			—	
Pyrostrand Test Winding				—

Figure 1. 18-inch Pyrostrand Furnace Installation

UNCLASSIFIED

Security Classification

## DOCUMENT CONTROL DATA - R &amp; D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)

Atlantic Research Corporation ✓

2a. REPORT SECURITY CLASSIFICATION

UNCLASSIFIED

2b. GROUP

3. REPORT TITLE

⑥ Development of High Temperature Resistant Materials for Use in Naval Ordnance

⑨ Quarterly Report, 6 Apr 1973 - 5 Jul 1973

5. AUTHOR(S) (Last name, first name, last name)

⑩ Bruno J. Macri

6. REPORT DATE

⑪ 18 Jul 1973

7a. TOTAL NO. OF PAGES

7b. NO. OF REFS

8a. ORIGINATOR'S REPORT NUMBER(S)

8b. CONTRACT OR GRANT NO.

⑫ NO0017-73-C-4306 ✓

⑬ TR-PL-10124-06-4 ✓

9. PROJECT NO.

49-5566

9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10. DISTRIBUTION STATEMENT

DISTRIBUTION STATEMENT A

Approved for public release;  
Distribution Unlimited

11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

Naval Ordnance Systems Command

13. ABSTRACT ✓

Work continued on the process scale-up to permit the fabrication of filament reinforced graphite composites in sizes up to 16-inch outside diameter. The vacuum chamber was constructed using stainless steel and has been received. The induction coil was fabricated and delivered the middle of May. Furnace components have been scaled-up and will be in operation before the next report period.

The scale up furnace includes several desirable features not available in previous fabrication equipment. These include external filament storage to permit the observation of the filament feed system during fabrication, three view ports to permit the visual observation of the fabrication process inside the furnace and arrangements to permit the use of the equipment for subsequent production requirements. A closed water system is being installed to permit furnace operation independent of city water. Individually controlled and monitored gas and water lines in and out of the induction furnace are provided.

DD FORM 1473  
1 NOV 65UNCLASSIFIED  
Security Classification

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14.	KEY WORDS	LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
	graphite composites pyrolytic graphite graphite yarn high temperature materials						

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Security Classification



Contract No. N00017-73-C-4306  
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